PERIÓDICO TCHÊ QUÍMICA
ARTIGO ORIGINAL

EXTRATO DE SEMENTES DE ABÓBORA COMO INIBIDOR DE CORROSÃO DE LIGA DE AÇO LEVE EM SOLUÇÃO ÁCIDA

THE EXTRACT OF PUMPKIN SEEDS AS A CORROSION INHIBITOR OF MILD STEEL ALLOY IN ACIDIC SOLUTION

JASIM, Ekhlas Qanber[1]; ALASADI, Erfan A.[1]; MOHAMMAD-ALI, Munther Abduljaleel[1*]

[1] Pharmaceutical Chemistry Department, College of Pharmacy, University of Basrah, Basrah, Iraq

** Corresponding author*
e-mail: munther.ali@uobasrah.edu.iq

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RESUMO

O problema da corrosão nas fábricas e instalações vitais continua sendo um dos obstáculos mais importantes que atrasam o progresso da produção e o aumento de sua quantidade. Para resolver o problema de corrosão, muitos inibidores inorgânicos e orgânicos têm sido usados. Recentemente, foram utilizados inibidores feitos de extratos vegetais, mais baratos e ambientalmente amigáveis. A inibição do extrato de sementes de abóbora na liga de aço leve à corrosão no meio ácido foi investigada por espectroscopia da impedância eletroquímica, técnica eletroquímica e método de perda de massa com solução aquosa estática ambiental. O efeito das concentrações de inibidores (10-50 mg/L) e o tempo de imersão (1 a 5 h) foram estudados na eficiência da inibição (η%) do extrato em aço leve (AL) imerso em uma solução de HCl 0,5 M. Todas as técnicas mostraram resultados muito bons nos valores do coeficiente de inibição. O percentual ótimo de η estava na faixa de 71 a 76%, dependendo do método utilizado. O tempo ideal foi de 5 horas. Os resultados das curvas de Tafel mostraram uma visão clara do comportamento do extrato, que atua como inibidor do tipo misto. Além disso, o teste de microscopia de força atômica foi aplicado para o estudo da morfologia da superfície da liga. Pelo exposto, o extrato de plantas de abóbora pode ser de grande benefício na inibição da corrosão na indústria.

Palavras-chave: *AFM, Corrosão, Impedância eletroquímica, Polarização potenciodinâmica, Sementes de abóbora.*

ABSTRACT

The problem of corrosion in factories and vital installations remains one of the most important obstacles that delay the progress of production and the increasing of its quantity. To solve the problem of corrosion, many inorganic and organic inhibitors have been used. Recently, inhibitors made from plant extracts, cheaper and environmentally friendly, were used. The inhibition of pumpkin seed extract on the corrosion mild steel alloy in the acidic medium was investigated using spectroscopy of the electrochemical impedance, Electrochemical technique and mass losing method with a static environmental aqueous acidic solution. Effect of inhibitor concentrations (10-50 mg/L) and immersion time (1–5 h) was studied on the inhibition efficiency (η%) of the extract on Mild Steel (MS) immersed in a 0.5 M HCl solution. All techniques showed very good matching results in the inhibition coefficient values. The optimum η% was in the range 71-76% depending on the method used. The optimum time was 5 hours. Tafel curves results showed a clear view of the extract behavior, which acts as a mixed-type inhibitor. Furthermore, the atomic force microscopy test was applied for studying surface morphology of alloy. From the foregoing, the pumpkin plant extract can be of great benefit in inhibiting corrosion in the industry.

Keywords: *AFM, Corrosion, Electrochemical impedance, Potentiodynamic polarization, Pumpkin seed.*

1. INTRODUCTION:

The acidic solutions may be used to remove the rust effect in different parts of the industrial processes. For example, diluted hydrochloric acid is used as broadly the pickling of steel alloys in oil and water pipes. Therefore, different types of inhibitors, which may be organic, inorganic, and natural extracts are widely used to reduce or control the dissolutions of metal (Karthiga *et al.,* 2015; Kliškić *et al.,* 2000).

The organic and inorganic inhibitors had very good anticorrosive activity, but they are toxic to both humans and the environment. These inhibitors may cause chronic or acute diseases because of damage in tissues of different organ systems or disturbing the vital function of metabolism or enzymic systems. In addition to these hazardous effects, this type of inhibitors is very costly and forced companies to spend millions on them that made the researchers instead of these synthetic inhibitors by naturally ones. Furthermore, this type of inhibitors are environmentally friendly, available sources, and cheap cost (El-Etre, 1998; Zhang *et al.,* 2015).

Up till now, many plant extracts have been investigated for their corrosion inhibitors behavior using acidic media on the mild steel. For example, *Murraya koenigii* (Yadav *et al.*, 2015; Sharmila *et al.,* 2010), *Terminalia chebula* (Patel *et al.,* 2009), *Carica papaya* (Kasuga *et al.,* 2018; Nwigwe *et al.,* 2019), *Nypa fruticans wurmb* (Orubite and Oforka, 2004; Michael and Olubunmi, 2014), *Emblica officianilis* (D'souza and Chattree, 2015; Saratha and Vasudha, 2010), and many other (El-Etre *et al.,* 2003). These studies including the effect of seed, leave or other parts extracts of plants as corrosion inhibitors on the mild steel in various concentrations of acidic media.

The inhibition behavior of *Murraya koenigii* (curry) leaves extract on the corrosion resistance of mild steel (MS) in nitric acid medium has been studied by gravimetric (or weight loss) measurements and scanning electron microscopy (Quraishi *et at., 2010)*. The inhibiting action of the fruit extract of *Terminalia chebula* (TC) on mild steel corrosion in 1 M HCl solution was studied using gravimetric, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) techniques. Other plant extracts have been investigated for their corrosion inhibitors behavior using acidic media on mild steel (Oguzie *et al.*, 2014). Many other studies, including the effect of seed, leave or other parts extracts of plants as corrosion inhibitors on the mild steel in various concentrations of acidic media (El-Etre, 2006; Zucchi and Omar, 1985).

The purpose of this research was to study the pumpkin seed extract as a corrosion inhibitor for mild steel (MS) alloy in acidic solution using electrochemical impedance, polarization of the potentiodynamic technique and mass losing method along with the surface morphology study of alloy using Atomic Force Microscopy (AFM).

2. MATERIALS AND METHODS:

2.1. Preparation of seed extract

Pumpkin seed that collected from the market of Basrah Old City was left to dry and ground to a fine powder. Twelve grams of this plant powder were mixed with D.W. (1000 mL) and subjected to reflux for about three hours. The resulting solution was passed through fine filter paper, and the clear solution was reduced to obtain 1 gram of extracted solid material of the original volume. The resulting new volume was utilized to test the corrosive inhibitor behavior by preparing different concentrations of aqueous solutions 10, 20, 30, 40, and 50 mg/L.

The coupons of mild steel (N80) was used to study the corrosion properties on a supplied from South Oil Company in Basrah that have the composition (wt.%): 0.80% Mn, 0.3% C, 0.22% Cu, 0.07% Ni, 0.05% P, 0.041%S and Fe is the remainder. The coupon was cleaned by smooth emery papers with grade

The coupons were rinsed with D.W. and subjected to degreasing by acetone and left to dry at room temperature, then kept in a desiccator until use. The concentrated HCl was used to prepare the solution of 0.5 M HCl by dilution with D.W. All the experiments were performed and repeated in the static solution.

2.2. Mass losing method

The experiments of mass loss were carried out on the mild steel alloy with a coupon dimensions (2.5 cm length) × (2.0 cm width) × (0.025 cm height) in a static solution of 0.5 M HCl with and without plant aqueous extract with diverse concentrations. The mass of each coupon was recorded by a digital sensitive balance with four decimal digital values and then immersed in 100 mL of acid solution with duration times 1 to 5 h at the temperature 25°C.

After every immersing time, the coupon was washed with distilled water, and the weighed again so as to calculate the efficiency of inhibition (η_{WL}) and the rate of corrosion (R_{corr}). For each run, all solutions were freshly prepared, and the temperature of the tested solution was controlled by a thermostatic auxiliary tool to give an accurate value.

Equation (1) used to calculate the corrosion rate ($R_{corr.}$) of mild steel (Batah *et al.,* 2017):

$$R_{corr} = \frac{\Delta W * K}{A * D * T} \quad \text{(Eq. 1)}$$

ΔW= mass losing of alloy (g), K= constant value (5.34x10^5), A= coupon area (cm^2), D= density of alloy (g/cm^3) and T= exposed time (h).

Equations (2) and (3) used to obtain the inhibition efficiency $\eta_{WL}(\%)$ and the degree of surface coverage θ_{WL}, respectively (Fouda *et al.*, 2017):

$$\eta_{WL}(\%) = \frac{W_o - W_i}{W_o} * 100 \quad \text{(Eq. 2)}$$

$$\theta_{WL} = \frac{W_o - W_i}{W_o} \quad \text{(Eq. 3)}$$

where, W_o and W_i (mg dm^{-2}) mass of the coupon after the dipping in acidic solution without or with extracted inhibitor.

2.3. Electrochemical measurements

The assays were performed at room temperature using three-electrode electrochemical cells containing a carbon steel working electrode with a 1 cm^2 surface area, a platinum auxiliary electrode, and a saturated calomel electrode (Reference electrode). The instrument used to obtain the curve of Tafel depends on the automatic changing in the potential electrode within the range from a positive value of 250 mV to the negative value of 250 mV assistant by scan rate of 0.5 mV per second of open circuit potential to investigate the inhibitory effect of the plant extract on the alloy of mild steel corrosion. The corrosion current densities (*i*) were utilized to draw the linear relationship of the Tafel segment. Then finally, the equation (4) used to calculate the inhibition efficiency $\eta_{pol}(\%)$ from the obtained values of current densities (*i*) (El Aoufir *et al.,* 2017; Abdul-Nabi and Jasim, 2014):

$$\eta_{pol}(\%) = \frac{i - i_o}{i} \times 100 \quad \text{(Eq. 4)}$$

where i and i_o are the uninhibited and inhibited corrosion current densities, respectively.

2.4. Impedance measurements

The impedance tests were investigated by AC signals of amplitude (10 mV) using the frequency spectrum range 100 kHz - 0.01 Hz. The values of charge transfer resistance (CTR) were obtained by Nyquist plots through the measurement of semi-circles diameter. The inhibition efficiency η_{EIC} (%) of the inhibitor was calculated from the values of CTR using equation (5) (Yang *et al.,* 2018):

$$\eta_{EIC}(\%) = \frac{R'_{ct} - R^o_{ct}}{R'_{ct}} \times 100 \quad \text{(Eq. 5)}$$

where R^o_{ct} and R'_{ct} represent the resistance of charge-transfer (RC-T) in the without and in with the diverse concentration of inhibitor.

The values of impedance from the plot were used to obtain the magnitudes of double-layer capacitance (C_{dl}) by using the equation (6) (Lgaz *et al.,* 2017):

$$|Z| = \frac{1}{2\pi f C_{dl}} \quad \text{(Eq. 6)}$$

2.5. Atomic Force Microscopy (AFM)

Atomic Force Microscopy (AFM) model CentaurU HR (Russia) was utilized in Physics and Energetic Faculty, Udmurtia, The Federal Republic of Russia. The alloy of mild steel that used as sections with the dimensions of (length 2.5 cm x width 2.5 cm and height 0.4cm) were subjected to dipping in the specific acidic solution using different concentrations of pumpkin seed extract (10, 20, 30, 40 and 50 mg/L) as corrosive inhibitors at 25^{o}C for 24 h. After this periodic time, the coupons were toke out, washed with D.W., left to dry, and then used for AFM tests.

3. RESULTS AND DISCUSSION:

3.1. Mass losing measurements

Table 1 and Figure 1 represent the output data of the study for the corrosion process of mild steel alloy in the acidic solution of 0.5 M using different amounts of pumpkin seed extract at 25^{o}C. The equations (1), (2) and (3) were used to calculate the corrosion rate (R_{corr}), the efficiency of inhibition ($\eta_{WL}(\%)$) and the coverage of surface (θ) values, respectively.

Table 1 and Figure 2 show the effect of concentration of plant extract on the inhibition efficiency, and the results indicate that by increasing the concentration the $\boldsymbol{\eta_{WL}}(\%)$ increases to reach the best effect of inhibitor at the concentration of 50 mg/L (Lgaz. *Et al.,* 2018). On the other hand, there is decreasing in the corrosion rate with increasing concentration, which gives a significant indication of the effect of the inhibitor on the corrosion process.

3.2. Tafel Polarization Measurements

The output data of the polarization measurements are listed in Table 2 for the corrosion of M-steel test in the 0.5 M acidic solution without and with different concentrations of pumpkin seed extract at the temperature of 25 °C. Figure 3 and Table 2 indicate that increasing the concentrations of pumpkin seed gives a significant decrease in reactions of H_2 reduction (cathodic process) and metal dissolution (anodic process). According to these results, the inhibitor gave the mechanisms of mixed type (Babic-Samardzija *et al.,* 2005; Jasim *et al.,* 2017).

The values of $\eta_{pol}(\%)$ and θ from pumpkin seed tests were calculated using Equation 4. Table 2 shows the determined values of I_{corr}, E_{corr}, Tafel slopes (βa and βc) and $\eta_{pol}(\%)$. The data indicated that there is an inversing effect between I_{corr} and the concentrations of pumpkin seed. The values of βa & βc remained almost unchanged with the addition of pumpkin seed concentrations, which gave a clear view that the adsorbed inhibitor decreases E_{corr} without affecting the reaction mechanism.

3.3. EIS measurements

EIS technique at OCP in a wide frequency range was used to study the capacitance at metal/electrolyte interface, which related to corrosion processes on the surface of mild steel coupons using the extract of pumpkin seed. Figure 4 shows Nyquist diagram for mild steel alloy dipped in 0.5M HCl solution at 25 °C using a series of concentrations of the extract as an inhibitor and without inhibitor at the certain open circuit potential.

From Figure 4, it's clear that there is increasing in the semi-circle diameter with inhibitor concentration rising. This result referred to the fact about an increase in corrosion resistance of the material. The optimum concentration of the extract was 50 mg/L, which gave Rp of 2100 reflected inhibition coefficient percentage of 76.19% as compared with the closed values calculated from Tafel and weight loss methods 74.41% and 71.93, respectively.

The opposite frequency values f_{max} can be used to calculate the magnitudes of electrochemical double-layer capacitance (C_{dl}) by utilizing equation (7) (Migahed *et al.,* 2016):

$$C_{dl} = \frac{1}{2\,\pi\, f_{max}\, R_{ct}} \qquad \text{(Eq.7)}$$

Where f_{max} is the opposite frequency when the imaginary component was maximum.

Table 3 referred to the EIS data which showed that the magnitudes of $\eta_{EIC}(\%)$ and R_{ct} is proportional to the concentration of inhibitor, whereas the C_{dl} values are proportional reversibly.

These results may be referred to the lowering of dielectric constant which effect on the rising in the thickness of the adsorbed electric double layer, which referred to the action of the inhibitor molecules as a thin layer to isolate the alloy from acidic solution by mix adsorption mechanism (Jasim *et al.,* 2015).

3.4. Morphology analysis by AFM

Figures 5-*i*, 5-*ii,* and 5-*iii* show the images of AFM for clear metal (control), the surface that subjected to corrosion by acidic medium (0.5M HCl) using or not the inhibitor extract. The pictures of the AFM gave a clear view of the smooth surface of the metal by using pumpkin seed extract concentrations inhibitor in 0.5M HCl. Table 4 gives the results of the AFM study represented by the values of *Ra* and *Rq*. The value of RMS, which equal to 55.2 nm, referred to the smoothing of the metal surface due to the adsorption of plant extract, which prevents the corrosion process as compared with the RMS value in the absence of inhibitor (120nm) (Elmsellem *et al.,* 2014).

4. CONCLUSIONS:

The pumpkin seed aqueous extract can be used to inhibit the corrosion of MS in the static acidic medium at 25°C. The optimum concentration of plant extract should be determined and utilized to give perfect protection efficiency. Using of plant extract in the industry as corrosion inhibitor enhances the green chemistry and environment friendly The pumpkin seed extract gave good corrosion inhibitor efficiency with closed values using different methods. Morphology analysis referred to the excellent inhibitory behavior of the plant extract.

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Table 1. *Effect of pumpkin seed extract on Dissolution Mild steel in 0.5M HCl*

Concentration (mg/L)	**ΔW (g)**	**Time (h)**	R_{corr}	$\eta_{WL}(\%)$	θ
0	0.0028	1	66.63		
	0.0062	2	73.77		
	0.0098	3	77.73		
	0.0134	4	79.72		
	0.0171	5	81.38		
10	0.0026	1	50.76	7.14	0.0714
	0.0041	2	40.02	33.87	0.3387
	0.0062	3	40.35	36.73	0.3673
	0.0080	4	39.05	40.30	0.4030
	0.0091	5	35.53	46.78	0.4678
20	0.0023	1	44.90	17.86	0.1786
	0.0039	2	38.07	37.10	0.3710
	0.0055	3	35.79	43.88	0.4388
	0.0072	4	35.14	46.27	0.4627
	0.0088	5	34.36	48.54	0.4854
30	0.0019	1	45.21	32.14	0.3214
	0.0033	2	39.26	46.77	0.4677
	0.0045	3	35.69	54.08	0.5408
	0.0056	4	33.31	58.21	0.5821
	0.0071	5	33.79	58.48	0.5848
40	0.0019	1	45.21	32.14	0.3214
	0.0030	2	35.69	51.61	0.5161
	0.0041	3	32.52	58.16	0.5816
	0.0050	4	29.74	62.69	0.6269
	0.0057	5	27.13	66.67	0.6667
50	0.0016	1	38.07	42.86	0.4286
	0.0028	2	33.31	54.84	0.5484
	0.0039	3	30.93	60.20	0.6020
	0.0046	4	27.36	65.67	0.6567
	0.0048	5	22.84	71.93	0.7193

Table 2. *The galvanostatic polarization results for mild steel*

Concentration of Inhibitor (mg/L)	I_{corr} **μA/cm²**	E_{corr} **mVolt**	***βc*** **mV/dm**	***βa*** **mV/dm**	$\eta_{pol}(\%)$
Blank	570	-221.2	-113.1	95.2	
10	334.46	-335.2	-103.1	87	41.32
20	263.97	-334.5	-96.5	72.3	53.68
30	209.18	-332.4	-95.2	86.1	63.30
40	184.61	-331.2	-138.9	75.5	67.61
50	145.83	-328.4	-140	70.7	74.41

Table 3. *EIS parameters for MS in the various concentrations pumpkin in 0.5M HCl*

Concentration (mg/L)	R_p	C_{dl}	$\eta_{EIC}(\%)$
0	249	639.50	
10	500	318.47	50.20
20	666	239.09	62.61
30	750	212.31	66.80
40	1000	159.23	75.10
50	2100	75.83	76.19

Table 4. *The morphology data by AFM*

Sample	**Average (Ra) Roughness (nm)**	**RMS (Rq) Roughness (nm)**
Clear mild (Control)	10.4	13.9
Mild steel immersed in 0.5M HCl	94	120
Mild steel immersed in 0.5M HCl + pumpkin	43	55.2

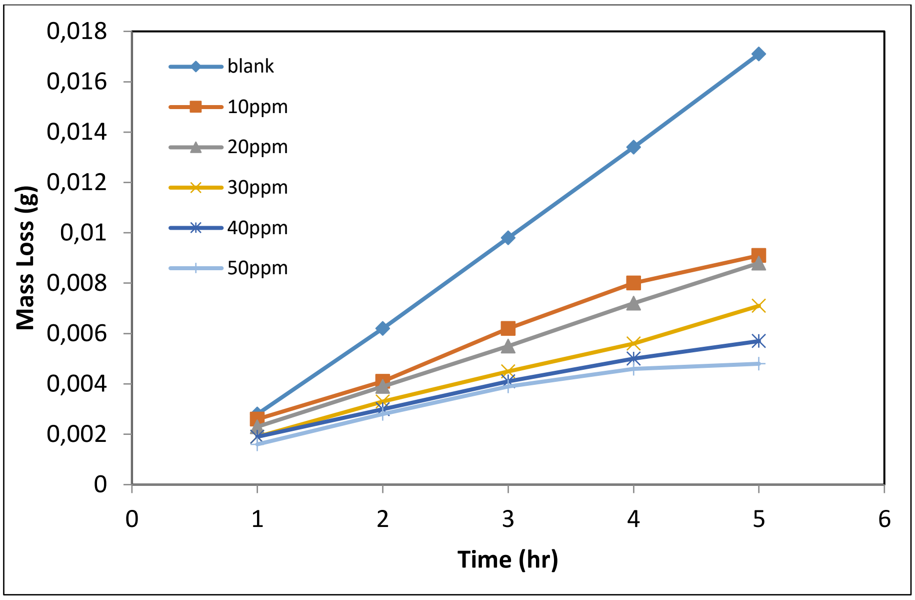

Figure 1. *Relationship between mass loss (g) of mild steel and time (hr) in 0.5M HCl using pumpkin extracts*

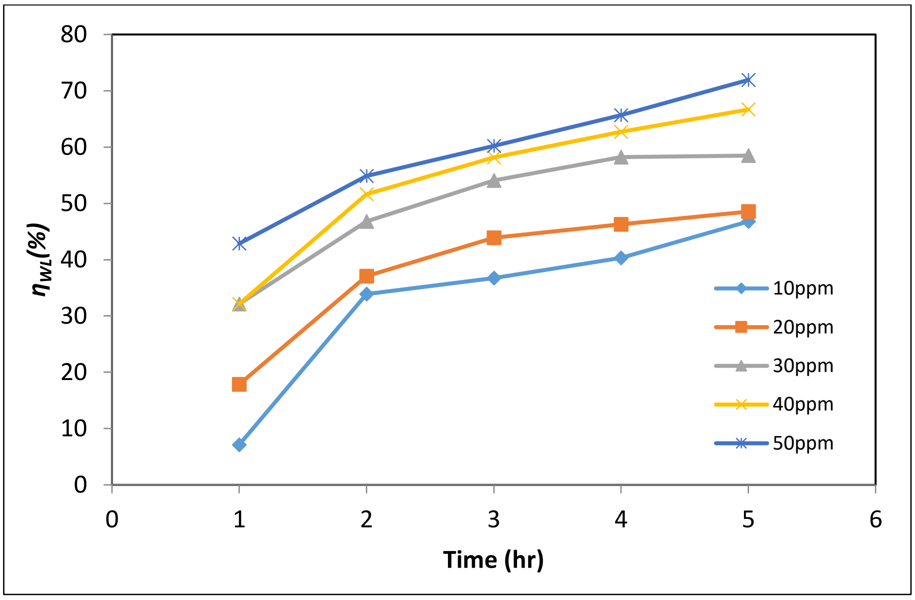

Figure 2. *Relationship between inhibition efficiency and time (hr) of mild steel in 0.5M HCl using pumpkin extracts*

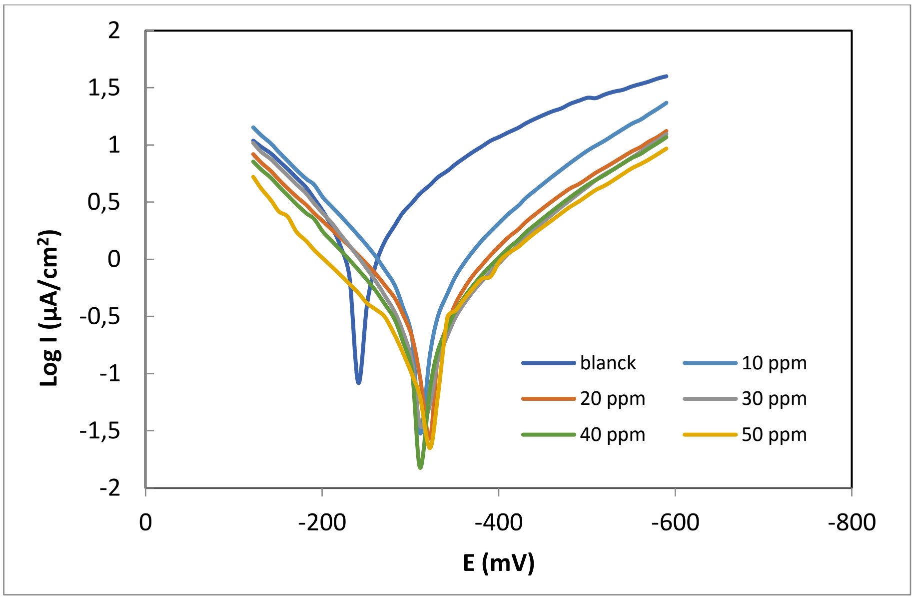

Figure 3. *Tafel curves of mild steel in 0.5 M HCl solution in the absence and presence of different concentrations of pumpkin inhibitor at 25°C*

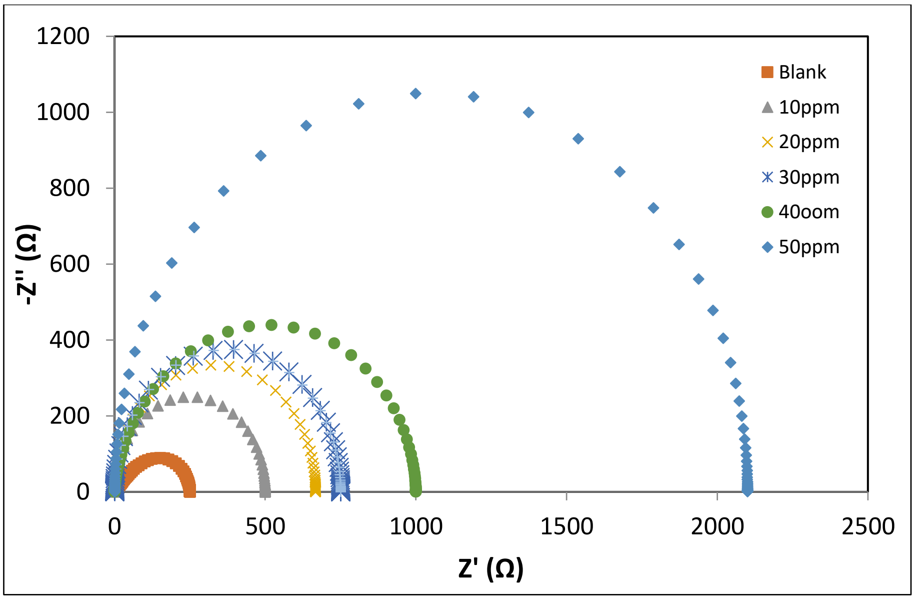

Figure 4. *Nyquist plots of mild steel in 0.5 M HCl with various concentrations of pumpkin seeds extracted at 25 °C*

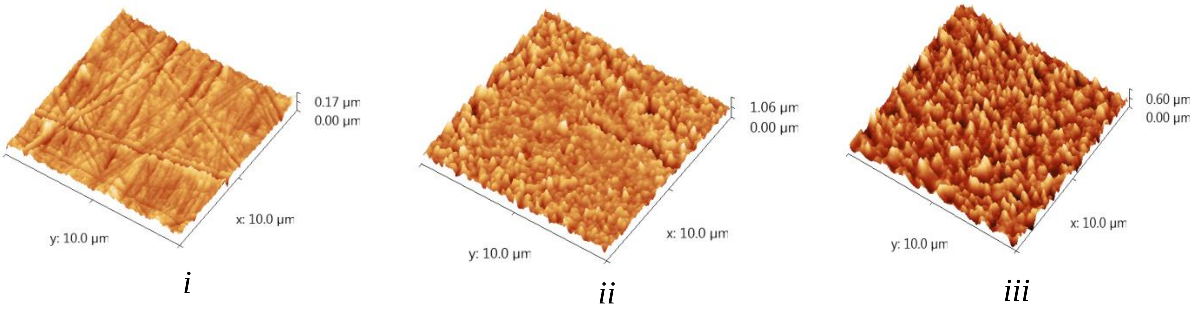

Figure 5. *The images of AFM of MS, i=Control, ii=immersed in 0.5M solution of HCl and iii= immersed in 0.5M solution of HCl + pumpkin seed extract*